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An Investigation of Viscoelastic Fracture Near and at Interfaces

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November 1988

Author:
W. G. Knauss

California Institute of Technology
1201 E. California Blvd
Pasadena, CA 91125

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FOREWORD

This final report was submitted by the California Institute of Technology, Pasadena, CA on completion of contract F04611-85-K-0106 with the Air Force Astronautics Laboratory (AFAL), Edwards AFB, CA. AFAL Project Manager was Jimmy Liu.

This report has been reviewed and is approved for release and distribution in accordance with the distribution statement on the cover and on the DD Form 1473.

CHI T. LIU
Project Manager

FRANCISCO Q. ROBERTO
Chief, Propellant Development Branch

FOR THE COMMANDER

CLARK W. HAWK
Director
Propulsion Division

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This report summarizes research on the effect of interfaces between two materials on the crack propagation process in its vicinity. The program contained both experimental as well as analytical components. The aim was to examine the applicability of optical caustics to crack propagation near interfaces. The main result of the work to date is the assurance that caustics can be employed to characterize interfacial cohesion and decohesion problems when crack tips are involved. On the experimental side of the program the method to generate bimaterial specimens with a plane interface has been developed and caustics involving large deformations have been studied and related to analysis. Examples of caustics in monolithic sheets of Solithane 113 of two different compositions (different material stiffnesses) are presented as well as caustics derived from crack tips at interfaces. On the analytical side the large deformation problem for two material interfaces between Mooney-Rivlin materials indicative of elastomeric behavior of solid propellant rocket materials has been studied numerically and found to be in (see over)

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Chi T. Liu	(805) 275-5274	RKPL

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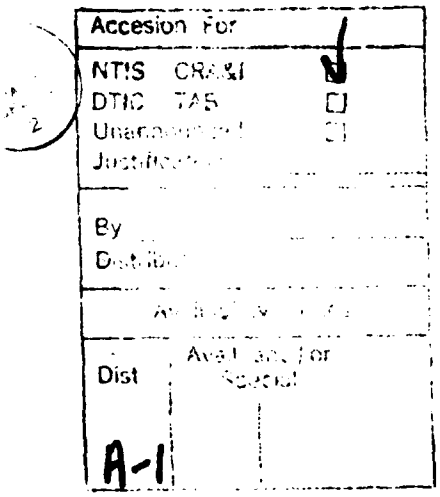
19. Abstract (Continued)

agreement with the crack tip. Moreover, the parameter controlling the description of loading conditions, a solution that is not possible on the basis of the above mentioned asymptotic relations. It has been found that this "large deformation stress intensity factor" is nearly linearly related to the far field loading - here applied as displacement boundary conditions - and that the displacement profile of the two crack surfaces have both the same characteristic shape but their magnitude is scaled by their respective domain stiffness.



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INTRODUCTION

There are many situations in the aerospace industry where parts that are joined by mechanical fasteners or adhesives are intended to act also as failure barriers. Aircraft skins fall in this category, since they are not intended to propagate cracks into neighboring panels nor into the supporting substructure. In solid propellant rocket motors the liner/insulation combination should be, ideally speaking, invulnerable to cracks that may start in the propellant. For, if such cracks penetrate the insulation or should separate the insulation from the motor case, burn-through of the motor case will result. These types of problems are not unique to the aerospace industry but occur in many branches of engineering. They occur in the automotive industry where parts are joined, mostly by (spot) welding as in the civil sector where, for example, gas transmission pipes must be joined such that cracks cannot propagate beyond certain pipe section lengths should a failure occur.

For metallic structures the associated design problems are dealt with effectively through fracture mechanics principles. This is true whether one considers joining with mechanical fasteners or with welded/brazed structures. In the latter case it is often possible to incorporate small sections of particularly tough material to act as potential "crack stoppers." For polymers joined adhesively the knowledge related to these kinds of problems is virtually non-existent. While we know that a particular design is fracture resistant if one employs "tough" materials we are neither sure of the precise meaning of "tough" in this context nor are we satisfactorily equipped to deal with the requisite stress analysis for fracture at or near interfaces. With respect to the question of "toughness" the uncertainty arises primarily from the complications associated with time dependent material behavior of the adherent materials. With respect to stress analysis problems we observe that while we know approximately what the stresses are we are not certain of the degree of approximation. This statement attains its real significance through the fact that crack growth rates are very sensitive to small variations in the stress levels. Practically speaking errors in the computed stresses of 5-10% may result in errors in estimated failure times on the order of factors of 10 or 100 (1-2 orders of magnitude) or more.

It is against this background that we started to investigate crack propagation near interfaces between viscoelastic solids. Basically two problems presented themselves and continue to be primary sources of uncertainty: One relates to the realistic definition of the stress and deformation state at the tip of a crack in such situations. The other one deals with the effect of two (or more) viscoelastic material functions on the crack growth behavior. Stated alternately, the second question might be phrased as a search for an understanding and prediction of the dependence on the material properties for crack growth between two viscoelastic materials if the crack growth behavior in a monolithic viscoelastic solid is understood.

In general, cracks can approach interfacial boundaries at various angles. Accordingly, the properties of the joined materials will affect the crack motion in different ways. The extreme situations are exemplified by cracks moving normal to the interface, on the one hand, and parallel, specifically along it, on the other. The change in the motion of the crack under these conditions will depend on whether the crack moves form the harder to softer material or vice versa.

In the case of solid propellant rocket motor design the adhesion and decohesion problem is of paramount importance for the following reason. In this area of engineering design the bonding surface is invariably near the case -the pressure vessel- where the insulation is bonded to the motor case. To this insulation is bonded the liner which forms the transition to the solid propellant. It turns out that, excepting possibly the transient stresses at the bore associated with ignition, the maximum stresses occur near the grain end or termination, in particular at the location of the insulation/liner so that special geometric

design precautions must be observed. Regardless of the latter, the case-near interfaces are often subject to high stresses and make the adhesion system vulnerable to damage. Because the insulation protects the case from the high temperatures of the burning gases it is imperative to maintain its integrity under all possible failure scenarios which includes the proclivity for a crack to propagate from the propellant through the liner to the insulation. While this scenario would result in burn-through of a steel case the future deployment of composite fiber reinforced cases which fail at considerably lower temperatures would be affected even more readily if only a thinning of the insulation occurred in such a failure process. In order to design the geometry of the liner/insulation system it is, therefore, important to understand the conditions that a) lead to interfacial or near-interfacial cracks and the conditions for their growth, and b) the path such cracks take when they propagate under generally applied stress fields removed from their fronts. This latter knowledge is important in order to determine whether a particular mechanical or thermo-mechanical loading introduced by the solid propellant grains likely to foster a detrimental propagation of a crack near an interface. To be sure, one attempts to design rockets in such a fashion that cracks do not appear at all; however, designing for the elimination of potential cracks is not at a stage where their absence can always be guaranteed and at times their appearance seems not to have even led to a mission failure. In such cases the extra assurance that a crack, undesirable by itself, propagates in a most benign direction is an important design and safety addition.

The mechanics of crack propagation near interfaces is a difficult subject for two reasons. First, it deals with basically the same material issues as crack propagation in monolithic materials, except that two materials are involved simultaneously. Thus a decision or criterion must be invoked that determines in which of the two materials the fracture will take place. Second, the analysis of the stresses and deformations, complicated in the present case of rubber-based compounds by large deformations, is a still debated issue; this latter issue arises apparently out of the linearization of the continuum formulation and is presently undergoing concentrated study by a number of investigators. The problem revolves around the appearance of oscillations in the stress field as the crack tip is approached. Associated with these stress field oscillations are displacements of the interfacial crack surfaces which also oscillate in such a way that the opposite crack faces would interpenetrate into each other; that is a physically unacceptable solution. This type of solution has been pointed out in the case of the rigid punch acting on a half space by Muskhelishvili in his treatise on Elasticity. It turns out that the domain in which these oscillations occur is on the order of 10^{-6} of the characteristic length of the problem which requires a crack or other size scale on the order of one to ten meters for the oscillations to be detectable with optical methods (wavelength of light = 5 micron). For solid propellants the size of particulates is on the order of 50 micron so that questions related to these oscillations appears hardly relevant.

This state of affairs with respect to the stress analysis of interface separation can be, apparently, resolved regardless of the scale of the microstructure if one allows for large deformations with respect to both material behavior and changes in geometry. Thus it appears more appropriate to deal with constitutive behavior that resembles that of propellants rather than that of steel or glass. Accordingly, we modeled the problems under investigation with large deformation and Mooney-Rivlin type material characteristics.

In order to study the motion of cracks near interfaces it is necessary to understand their motion through a monolithic solid of either properties. For this purpose one needs to measure the rate of crack speed in viscoelastic solids of either of the two materials. This study is presented here. Moreover, and possibly more importantly, to study crack growth near interfaces in a macroscopically measurable scale it is necessary to produce specimens which allow for a planar interface so that standard analytical tools may be brought to bear on the data analysis. The process to develop this specimen procedure is also presented here.

Finally, the tool to determine or at least check on the conditions at the tip of an advancing crack it is necessary to develop an experimental tool to assess the stresses at the propagating crack tip. The method chosen for this purpose was that of caustics. This choice was made primarily because this method is relatively simple to apply and to analyze quickly, so that "on the spot" evaluation during the running of the test can be made. This method turns out to be viable as long as the zone from which the data is taken for the generation of the caustic is not too small.

CRACK PROPAGATION IN MONOLITHIC VISCOELASTIC SOLIDS

It should be recalled that a major purpose of this study is the development of an understanding of what controls the speed and direction of cracks near interfaces. Regarding the question of speed it is known that in monolithic, linearly viscoelastic solids the speed of a crack is governed, to first order, by the instantaneous stress intensity factor and by the creep function of the solid Reference [1]. It has also been argued Reference [2] on the basis of the same theory that when a crack propagates along an interface between two viscoelastic solids then the average of the creep functions for the two solids replaces the single creep function. No experimental verification of this proposition has been performed nor attempted, to our knowledge, up to this time.

We delineate first measurements of crack propagation in those materials that are to constitute the adherents in the later study of crack propagation involving viscoelastic bimaterials. In performing these experiments we are interested in ascertaining

a. The experimental relation between crack tip conditions (stress intensity factor) and the speed of crack growth in a single viscoelastic solid (in sheet form).

b. Perform the measurements under) for both adherends of different viscoelastic response. One of these (65/35 composition) exhibits more viscous behavior than the other (50/50 composition); for definition of composition see later description below.

c. Ascertaining whether these measurements correspond to the predictions offered by the linear theory of viscoelasticity.

d. Provided c) is verified, ascertain whether the dual material problem for interface parallel crack propagation agrees with the predictions derived from linear elasticity/viscoelasticity.

In approaching these objectives we make partial use of previous results on one of the materials to be used, Solithane 113. Solithane is the trade name for a polyurethane elastomer manufactured by the Thiokol Chemical Corporation. It is furnished in two components, a so-called Urethane Resin and a Catalyst. By mixing the two components in various ratios the mechanical properties of the final elastomer can be varied over a considerable range. Depending upon the care with which moisture is prevented from influencing the chemical reactions, the elastomer is nearly colorless and transparent, or slightly amber colored if moisture has entered the system. Prior to curing (at 165°C for the material in this report) the mixture is easily cast at 60°C.

Chemically the "Resin" is a trifunctional isocyanate which is the product of a reaction between Castor Oil and Tolylenediisocyanate (TDI). Urethane crosslinks are then introduced between the "Resin" chains by adding the "Catalyst" and curing the mixture at an elevated temperature. The "Catalyst" is a triol and consists essentially of Castor Oil. The commercial designations of the "Resin" and "Catalyst" used for the production of Solithane 113 are Thiokol Solithane 113 Urethane Resin and Thikol Urethane Resin Catalyst C113-300 Urethane Resin and Thikol Urethane Resin Catalyst C113-300.

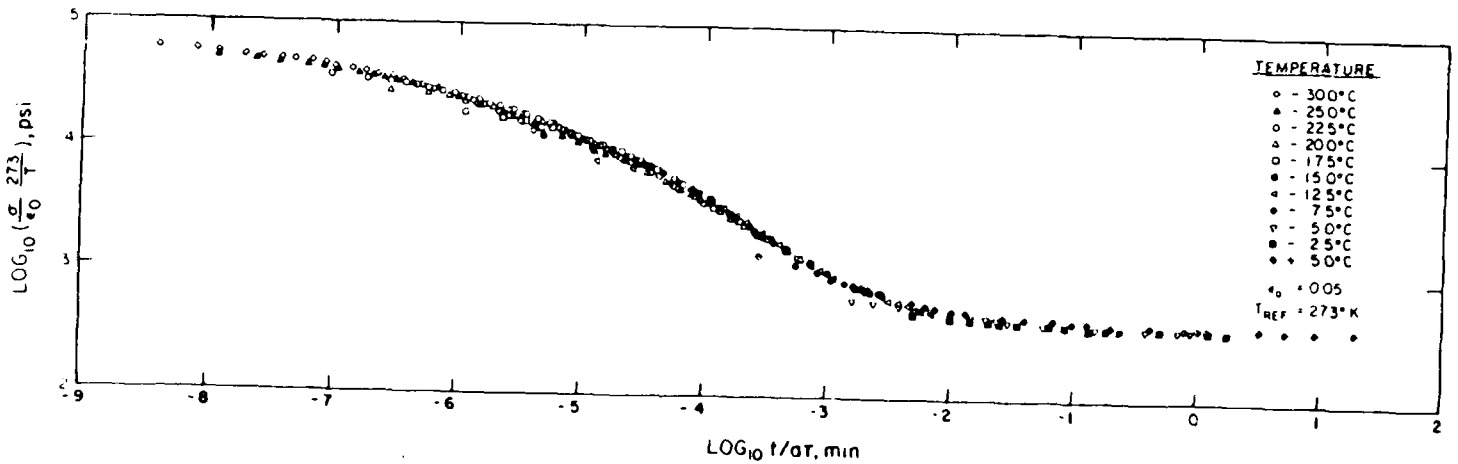


Figure 1. Master curve for temperature reduced relaxation modulus of unswollen Solithane 50/50, reference temperature 0°C.

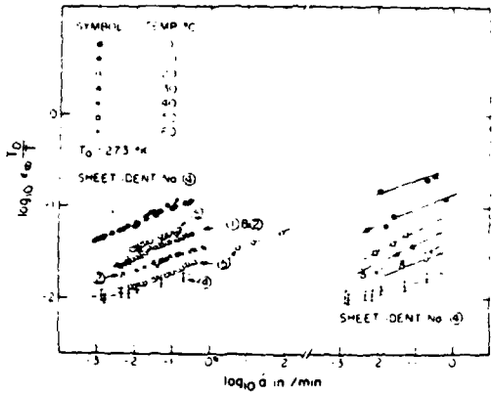


Figure 2a. Crack-propagation rates at different strains ε_∞ and different temperatures. Left set of data from various sheets; right set from one sheet only to assess the effect of sheet-to-sheet variability in properties. From Reference [1].

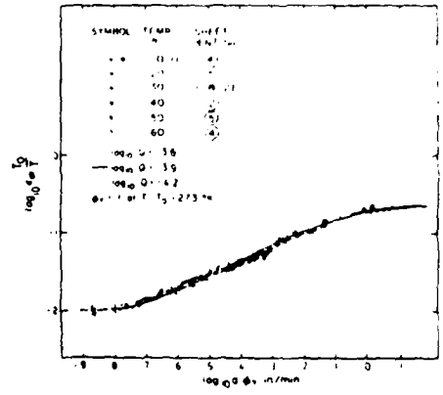


Figure 2b. Master curve of crack-propagation rates obtained on various sheets. From Reference [1].

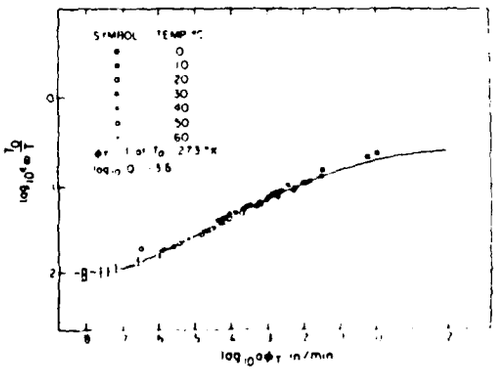


Figure 2c. Master curve of crack-propagation rates obtained on specimens cut from a single sheet. From Reference [1].

In an earlier study Reference [3] the physical properties including crack propagation behavior of the 50/50 composition had been examined. While these data, shown in Figures 1 and 2a-c, were obtained some time ago. We were, for initialization purposes, willing to assume that these data were still valid. Such an assumption is not necessarily a foregone conclusion, since often suppliers of chemicals such as the ingredients of Solithane 113 make changes in the formulation which is not evident until detailed tests are performed. Such uncertainties notwithstanding we concentrated on first examining the companion 65/35 material with respect to crack speed behavior.

Such a study seemed necessary in order to determine separately for reference purposes the behavior of the two materials involved. It turned out -incidentally as before with the 50/50 composition - that this characterization is not trivial but requires considerable effort in careful, detailed and repeated measurements. The difficulty results primarily from the fact that crack propagation speeds are a very sensitive function of the stress intensity factor. Many measurements may have to be made before it can be recognized that some small experimental variable is not set or measured properly.

Although some data on relaxation behavior was available on the second formulation planned for use in this study, namely the composition of 65/35 resin/catalyst ratio, no crack propagation measurements on this material had ever been conducted. The 65/36 composition is considerably more viscous or dissipative than the 50/50 composition, though their long term moduli (rubbery moduli) are very closely the same. Figure 3 shows a comparison of the relaxation behavior of these two solids as taken from Reference [3].

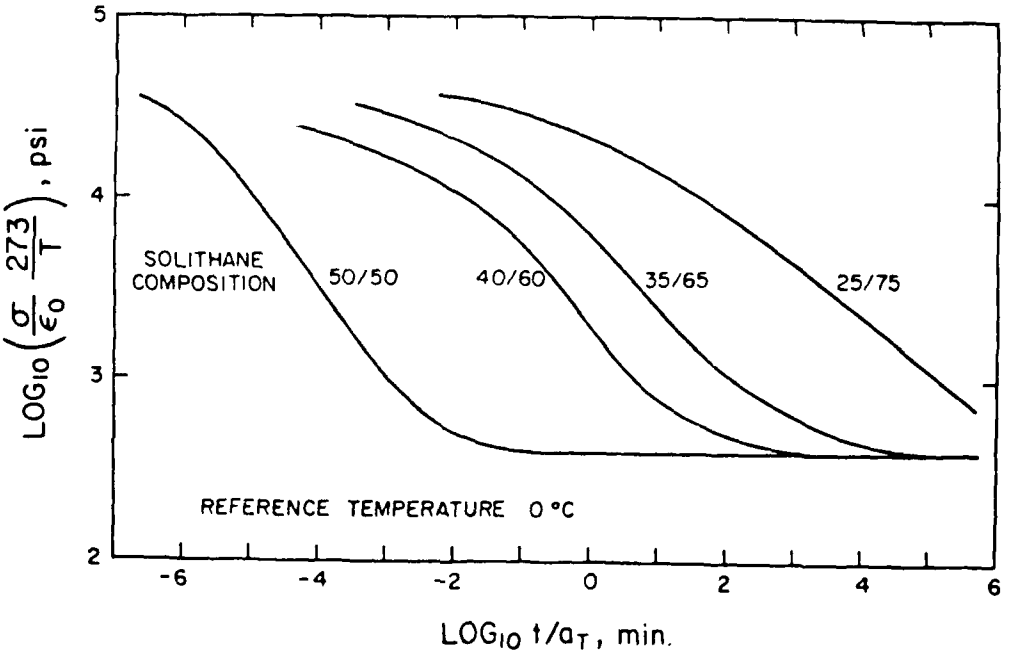


Figure 3. Relaxation curves for various compositions of Solithane 113.

CRACK SPEED CHARACTERIZATION IN THE 65/35 COMPOSITION

The crack propagation measurements were made on specimens of the type shown in Figure 4. Straining resulted from displacing the two long rails apart in a parallel manner so that in the central portion of the strip a homogeneous stress field resulted in which the crack could propagate at a constant rate because the crack tip conditions remained constant. These conditions are indeed constant as long as the crack tip is away from the sides or ends of the specimen by about or more than a strip height h. Thus multiple measurements of crack speed could be made if the crack was allowed to propagate at any one speed for about 3/4 to one inch. It occurred repeatedly that fracture started prematurely from a (right) corner where the Solithane sheet material was bonded to the rails; this feature had not been a problem before in testing the 50/50 composition. However, in the present case this problem was alleviated by cutting the non-cracked side of the specimen in the manner shown as the dotted line in Figure 4.

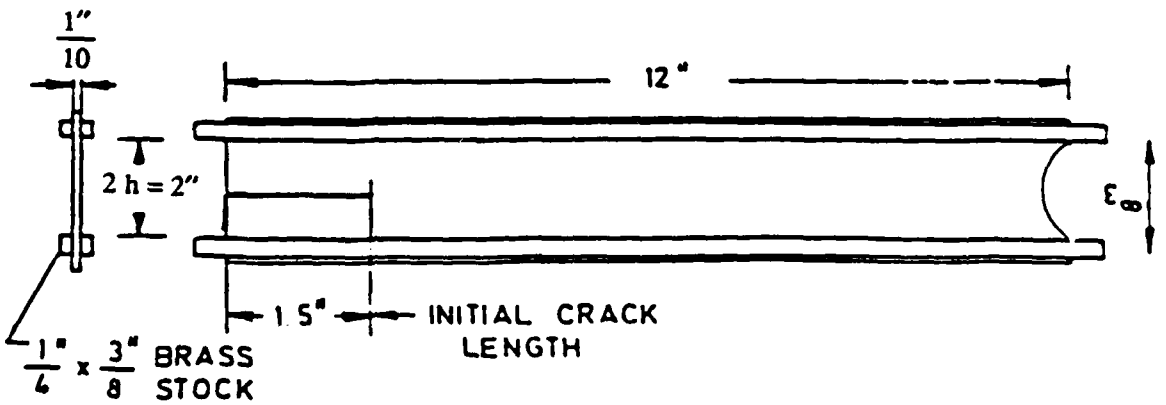


Figure 4. Test specimen for crack propagation tests.

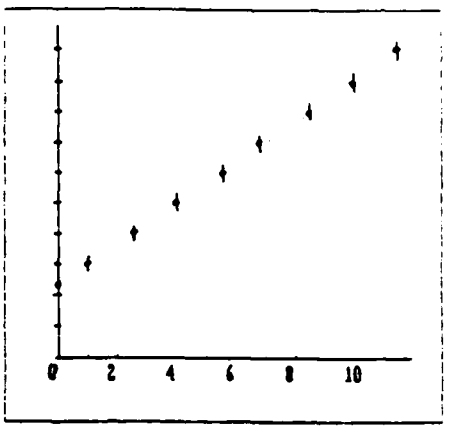
For each given strain on the order of ten measurements of the crack tip position and the corresponding times were made. These data were then computer-reduced by fitting a least squares straight line to them and performing the Chi-square test on the data. In addition to each plot the deviation of the crack tip position from the straight line fit was plotted to give a quick visual indication of the accuracy of the measurements. A typical example of such a data reduction plot for each velocity is shown in Figure 5. These types of measurements were repeated at five temperatures from 40 to 70°C.

Around 800 measurements were made of which about one third were found to lead to inconsistent results. The reason, most likely, was that the zero strain was not established correctly. In order to determine the strain accurately it was necessary to set the displacement of the straining device for the specimen in Figure 4 such as to ensure the zero strain state. This was accomplished by determining the situation when the crack tip was free of strain as determined photoelastically. That determination was probably the most critical parameter in the testing process.

Figure 6 shows a plot of crack propagation speed as a function of the temperature reduced strain, which is proportional to the stress intensity factor, at several temperatures. The applied strain ε_o is proportional to the stress intensity factor K through the relation

$$K = \frac{E_\infty \varepsilon_o \sqrt{h/2}}{\sqrt{1-\nu^2}} \tag{1}$$

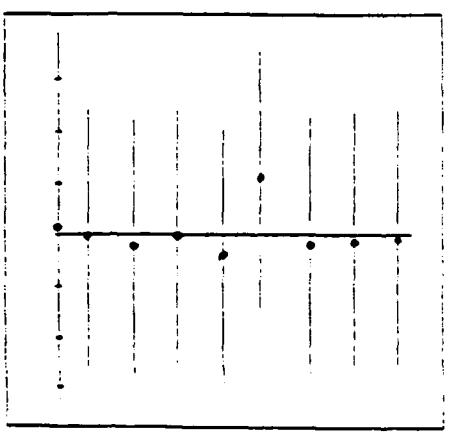
with h the specimen height. In the present work we assume that enough time has lapsed between strain application and the crack propagation measurements so that the material has relaxed to its long time equilibrium behavior. Inasmuch as the long term modulus E_∞ is, according to the classical theory of rubber elasticity, proportional to the absolute temperature we apply that temperature reduction here to the strain for the purpose of constructing the crack propagation master curve. This data appears shiftable according to the normal time-temperature superposition principle and the resulting "master curve" is shown in Figure 7. When one compares this data with that for the 50/50 composition one notices that crack speed at comparable strains is less for the 65/35 composition than for the former one. This result is as expected on the basis of the relaxation behavior shown in Figure 3 and of the theoretical results presented in Reference [1].



exp66
XOFF = 0
DX = 1
YOFF = 9.8
DY = .1

Y = A+B*X
(N = 9)
A = 10.03367
SIG(A) = 1.479745E-02
B = 6.600804E-02
SIG(B) = 2.21827E-03
CHI^2/(N-2) = 3.407705E-02

Experiment 66 50°C .058in.



exp66
XOFF = 0
DX = 1
YOFF = 0
DY = .01

Figure 5. Example of data analysis for determining crack speed (speed = B in in./min.).

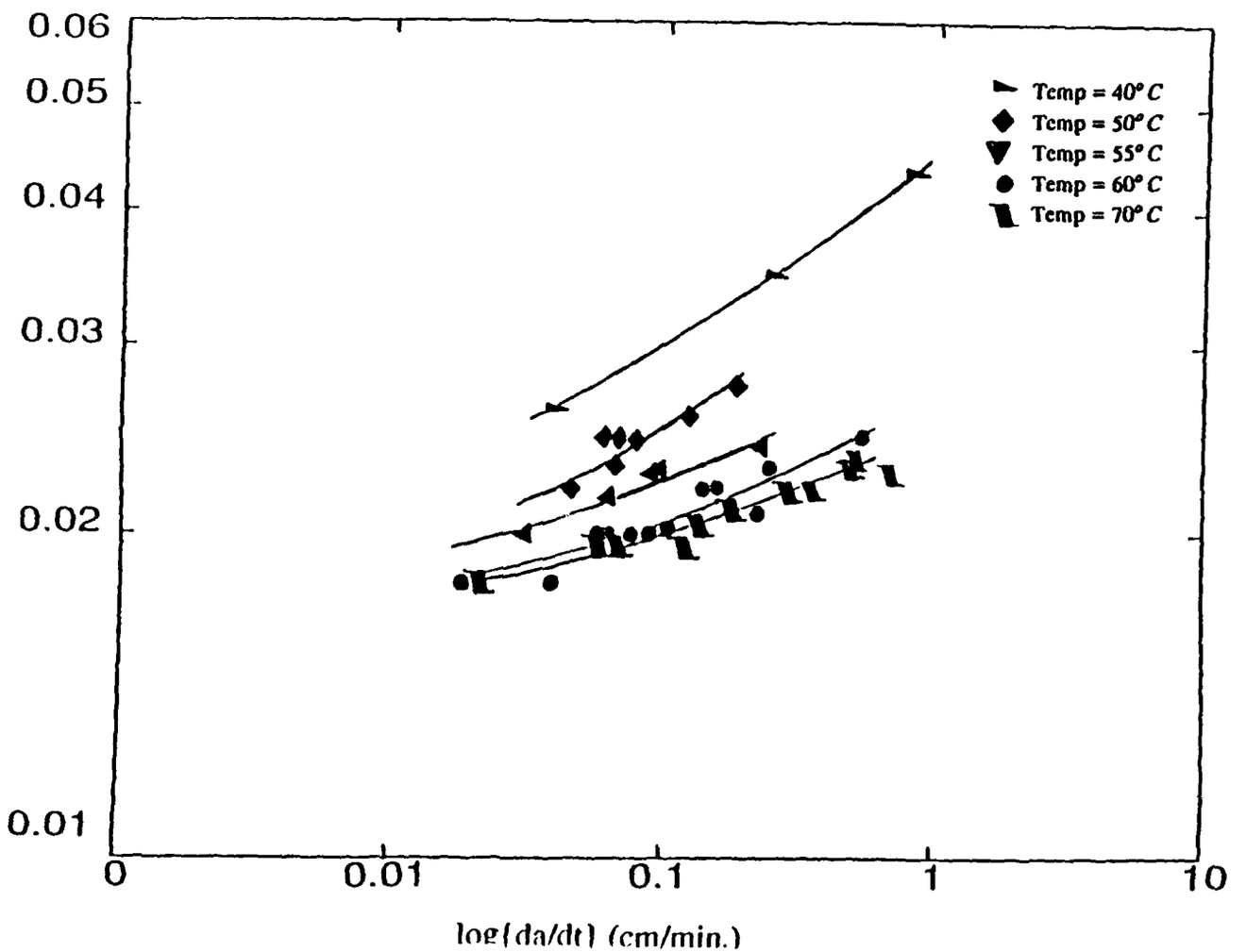


Figure 6. Crack velocity in the 65/35 composition as a function of temperature reduced strain.

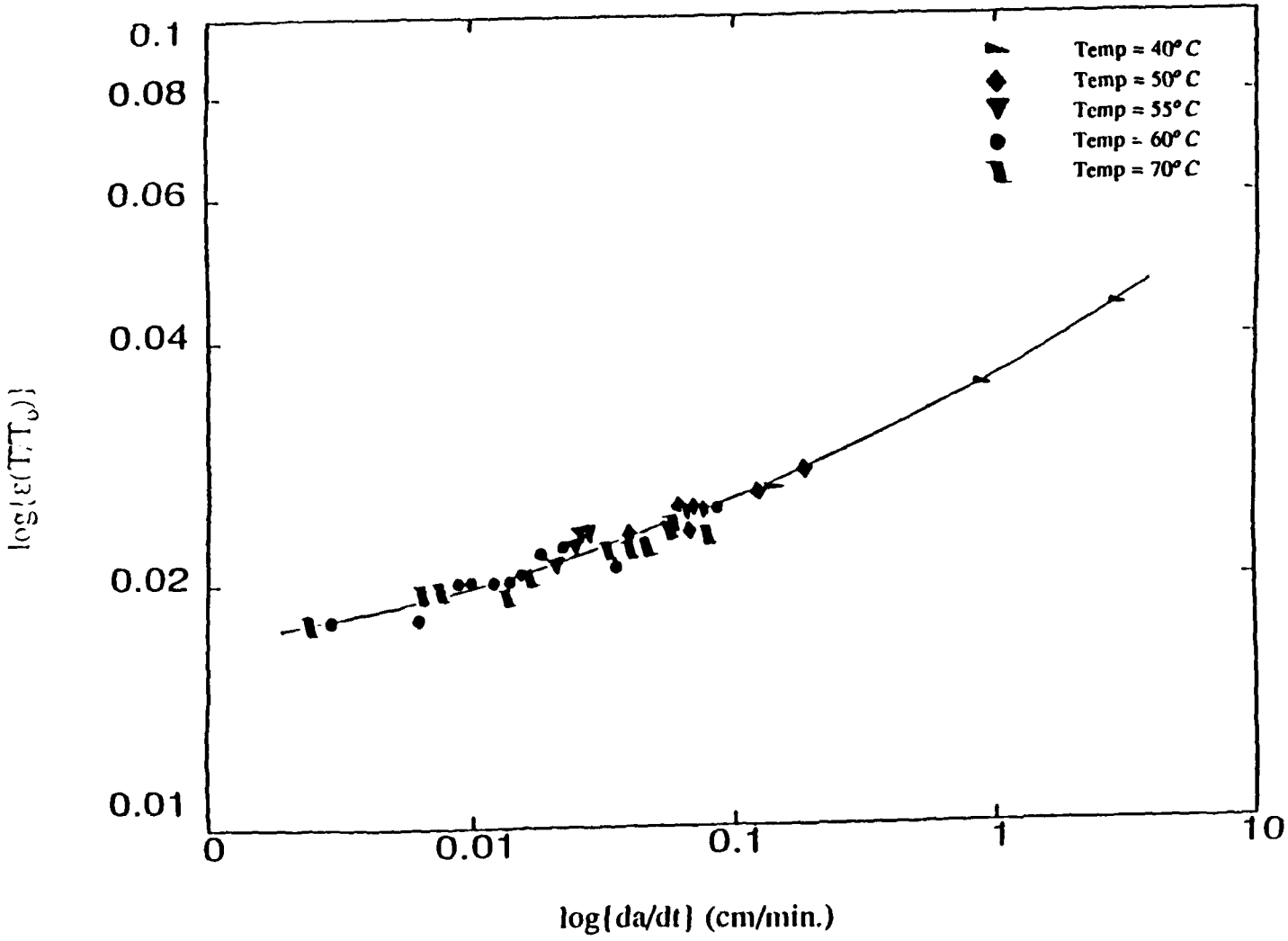


Figure 7. Master curve of crack speed desired from Figure 6.

DESIGN OF THE INTERFACE FRACTURE SPECIMEN

In order to study fracture at interfaces it is necessary to provide specimens which allow for a well defined interface. In addition, for analytical purposes it may be absolutely necessary to deal with an interface that is described by a simple topology. The simplest configuration is that of a plane which is, actually, necessary for most analytical purposes. It turns out that the production of two-material specimens having a flat interface is a surprisingly difficult undertaking.

Normally monolithic sheets of Solithane 113 are cast in a mold made up of two 3/4 inch thick aluminum platens which have grooves cut into their flat surfaces to attach, via vacuum, highly polished Ferrotype plates, such as used to produce high gloss finish on photographic paper. Two such "armed" platens are then bolted together, suitable separated by a spacer to render sheet material of a given thickness. Holes are placed into the platens for filling the mold and for allowing escape of air during molding. Actually, the mold is filled by sucking the Solithane into the mold cavity through the application of a vacuum.

In order to produce bi-material sheets it was first tried to fill the mold partially (half way) with the mold faces aligned vertically; that portion of Solithane was then cured lightly so that mixing with a second charge of different composition could not take place. It developed that a meniscus formed in the process of the first half-casting; this meniscus was was too pronounced to yield an even approximately flat interface as viewed across the thickness coordinate of the sheet.

Specimens with this meniscus interface were useful only in studies in which the crack approached the interface at some angle on the order of 90 degrees. In that event the details of the interface were less important than the thickness averaged effect of the interface on the crack tip stress field. For studies in which the crack was expected to move parallel and close to the interface such an interface topology was clearly not permissible.

A second method for producing two-material specimens was to attempt interfacing 65/35 sheet stock with a pre-cut flat edge with a casting of Solithane 50/50. To this end half cured 65/35 sheet specimens were produced which were removed from the mold with some difficulty because of their fragile nature. Using sharp razor blades the sheets could be cut into strips with very smooth edges which would provide a good flat interface if the other composition could be successfully bonded to it.

However, here the differential thermal expansion and the cure shrinkage of Solithane 113 provided difficulties: When the Solithane is removed from the mold it shrinks because of both thermal cool-down and because of volume decrease resulting from the cure process; the latter amounts to about 4% volume decrease. Upon placing the strip or half sheet of Solithane after cutting back in the mold it cannot quite fill the thickness of the cavity. As a result the newly added alternate composition flows past the partially cured material and produces surface blemishes which make the sample unsuitable for optical evaluation. Moreover, even when these surface blemishes were reduced by redesigning a second mold, it turns out that the final composite sheet warped upon removal from the mold because the first composition had already undergone cure shrinkeage while the second addition had not, so that the latter had to contract more and introduced residual deformations and stresses.

A problem arose with the multiple and intermittent use of the mold's highly polished surfaces. Because of the multiple experimentation these became scratched and the Ferrotype plates used for the mold are no longer manufactured. For this reason a new mold made of 1/2 inch thick glass plates was constructed, and the spacer formerly made of Teflon, was replaced by spacers of the more

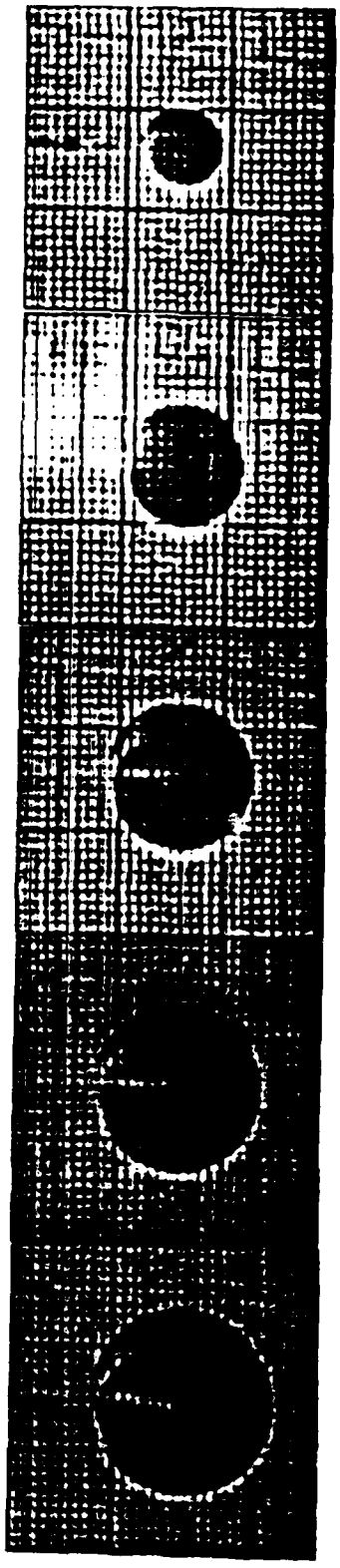
compliant Solithane in order to allow for the shrinkeage of the first casting. However, while the mold performs well in principle it was virtually impossible to make the glass plates stick to the first-cast Solithane over its entire surface. As a result the surface properties were unsuitable for optics work.

Production Process for Making High Performance Interface Specimens

The method that produces interface specimens requires that the first-cast material not be removed from the mold until the second-cast material has solidified. It still requires two cure cycles for the first-cast solid. Glass plates were used for the mold. The basis for the operation is the use of Silicone rubber as dam material against which Solithane can be cast.

First a mold was made with General Electric RTV-630 silicone rubber. The Silicone rubber was cast against a finely machined plate of aluminum and of the appropriate sheet thickness. These strips were arranged to form a four inch wide cavity in the mold into which the first cast of Solithane could be entered. After precuring this cast the Silicone rubber could be removed by pulling it out from between the mold plates without disturbing the adhesion of the Solithane to the glass or destroying the critical surfaces of the Solithane. Upon injecting the second Solithane formulation it would form an interface with the first cast that was completely planar as determined by inspection without any special optical tools. There is still a bit of a problem in that the first cast Solithane, 65/35 in these trials, tended to stick more to the glass mold surfaces than the 50/50 formulation; this is perhaps a matter that can be resolved by optimizing the pre-cure process more cafefully.

This casting process provides then two interfaces which were, because of the spacing of the Silicone dams mentioned above, four inches apart. Thus each casting can produce two strip specimens two inches wide and containing an interface in the middle as shown in Figure 11.



Caustics curves for 50/50 Solithane

u=.020"
ε=1%

D=8.0mm

u=.040"
ε=2%

D=12.0mm

u=.060"
ε=3%

D=15.0mm

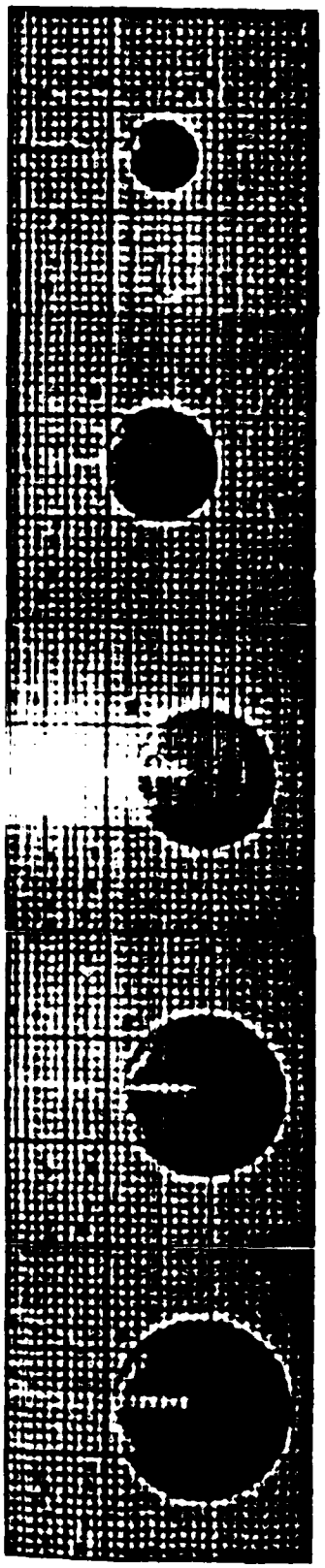
u=.080"
ε=4%

D=16.5mm

u=.100"
ε=5%

D=18.0mm

Figure 8. Caustics in the 50/50 composition for various strains.



Caustics curves for 65/35 Solithane

u=.020"
ε=1%

D=7.0mm

u=.040"
ε=2%

D=11.5mm

u=.060"
ε=3%

D=14.0mm

u=.080"
ε=4%

D=16.0mm

u=.100"
ε=5%

D=17.5mm

Figure 9. Caustics in the 65/35 composition for various strains.

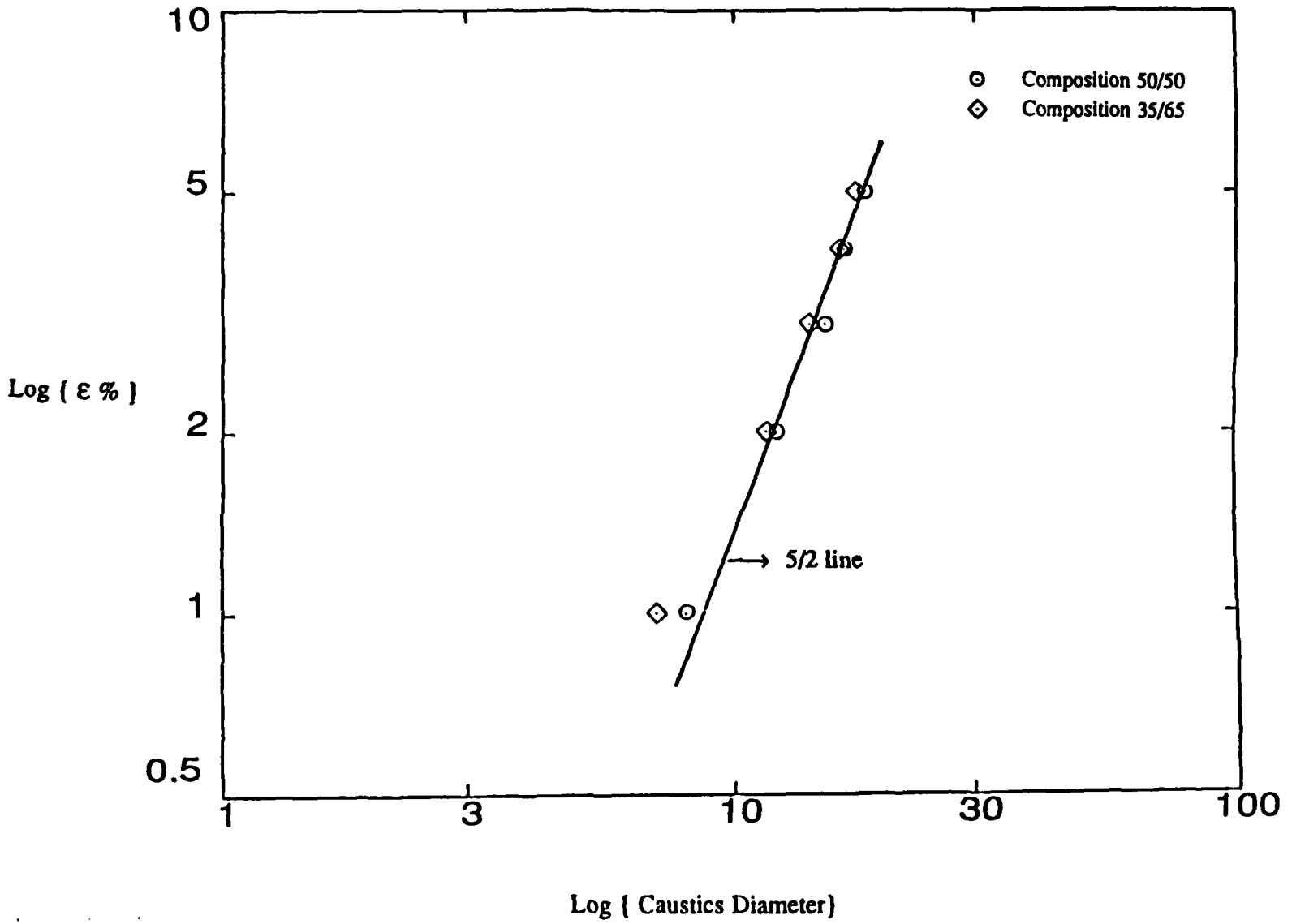
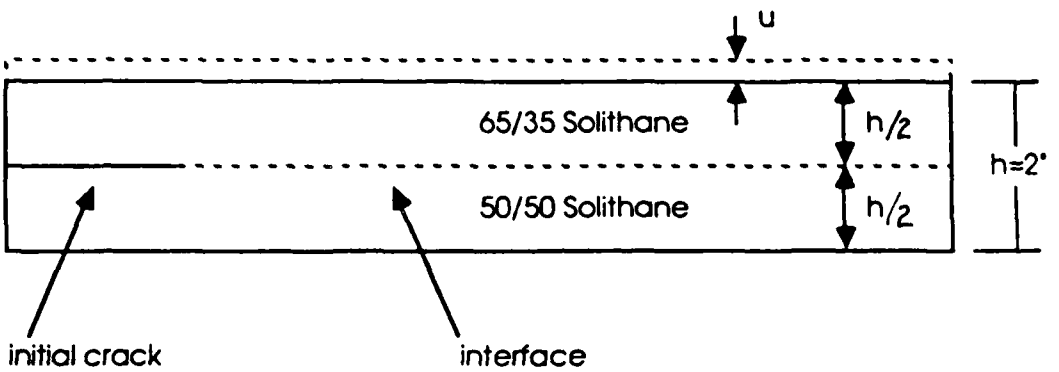


Figure 10. Caustic diameter as a function of a strain deduced from Figures 8 and 9.



u=displacement
Specimen has thickness, d.

Figure 11. Bi-material specimen.

EXPERIMENTAL CAUSTICS NEAR INTERFACES

A number of experiments were conducted with caustics in specimens containing an interface, as well as with cracks placed in single or monolithic material sheets. The caustics in monolithic materials can be used to determine the experimental parameters to characterize the optical properties of the Solithane compositions and it was of interest to check whether the caustics had indeed the shapes expected on the basis of linear and nonlinear analysis.

In the present experimental set up it was difficult to guarantee the absence of any shear deformation parallel to the crack. The photoelastic set up which allowed the determination of zero strain before loading was commenced was not particularly sensitive to shear indications.

Figures 8 and 9 show typical sequences of caustics in monolithic compositions of Solithane at various strain levels pertinent to the test geometry in Figure 1. One notices in both sets that at small strains the caustic is not perfectly symmetric, indicating that some shear or in-plane antisymmetric deformation is present. As the strain is increased the symmetric deformation begins to dominate, so that in both cases the caustic at 4 and 5% strain are essentially typical of symmetric loading.

If one plots the major diameter "D" of the caustic as a function of the applied strain - which is very nearly proportional to the stress intensity - one would, by linear theory, obtain a power law relation with exponent 5/2. Figure 10 shows this relation in log-log fashion along with the straight power law line of slope 5/2. We note that at the higher strain levels that line is followed quite well though not at lower strain levels, and it remains to be seen whether this observation represents a systematic deviation or an error in the measurement at low strain where relative errors tend to be larger. It should be noted that with the normal accuracy associated with most caustic work - often uncertainties on the order of +/- 10% occur - this result is quite acceptable.

In the appendix we show a typical caustic for an interface crack in comparison to the computed counterpart. It turns out that the caustic changes shape considerably, but that is to be expected in view of the observation in the appendix that the deformation of the crack surface and thus perhaps that of the region around the crack tip follows quite closely the compliance of the two materials.

LARGE DEFORMATION ANALYSIS OF CAUSTICS FOR INTERFACE CRACKS

The determination of stress and deformation fields around the tip of a crack located at and parallel to the interface is of primary importance in a study of this kind. Because of the asymmetry in the material distribution we expect the typical result that for crack-normal loading far from the tip the stress and deformation fields around the tip are also not symmetric with respect to the interface. That expectation is clearly fulfilled, and as a consequence the caustic exhibits the typical characteristics of both mode I and mode II deformations.

The basic question at issue here is whether the large deformations encountered in connection with rubbery solids can still be characterized in terms of caustics in a meaningful way. The overall answer is affirmative. The reason for this affirmation is basically that the nonlinear deformations are confined to a very small domain around the crack tip and that the region from which the caustic characterization is drawn falls into a larger domain which is governed by nearly linear material and deformation response. This is a fortunate situation because it allows one to address much of the present kind of problems within the realm of linearized theory.

The developments leading to this statement are summarized in a study contained in the appendix. This work has been presented in September 1987 at the Society of Engineering Science in Salt Lake City, Utah and is slated for publication in the Journal of that Society. For this reason it is felt that inclusion of the completed manuscript in this report as an appendix, rather than a separate, essentially repeat account, is appropriate.

An important result of this analysis is that the asymptotic large deformation solution passes over smoothly into the finite domain surrounded by the crack tip and analyzed here numerically. For many strains ε_o experienced in mechanical designs, i.e. on the order of 15%, the crack tip stresses are related nearly linearly to the far field loading (here in the form of far field straining) though the distribution of stresses is different from that rendered by linear elasticity solutions. Furthermore, the deformations of the crack surfaces (crack profile) are **independent of the ratio of the material stiffnesses** though the magnitude of the crack face displacement depends on the stiffness of the material to which the particular crack face belongs. These are important results because they considerably simplify analyses dealing with interface failure.

It should be pointed out that the computations for the caustics associated with the interface deformations were carried out for the case of reflection caustics. For analytical purposes it appeared better to consider this particular case rather than deal with transmission caustics (for further detailed results the reader is referred to the appendix) which depend to a large degree on the specimen thickness and the optical stress properties of the materials.

Since optical properties for the experimental materials involved had not been determined it seemed prudent to restrict consideration to the reflection caustic which derives from only the surface deformations without reference to the properties of the underlying material. In order to characterize the optical transmission caustic it is necessary to either determine certain optical constants or functions of the material(s) involved or to deduce such properties from caustics observed in sheet material of well defined thickness. It is with this idea in mind that experiments were initiated on caustics around crack tips in the two monolithic compositions. This effort has been summarized briefly in the section entitled "Experimental Caustics Near Interfaces".

Mention should be made of the fact that in a laboratory specimen the three-dimensionality of the deformation around the crack tip at the interface generates portions of the caustic that reflect the difference in the stiffness of the two adherends in the out-of-plane deformations. In the analytical work it was

assumed that plane stress conditions prevailed. In detail that means that along the interface there would be a step displacement as one travels across the interface. In reality there would be a (locally high) displacement gradient which is thus not modeled. The consequence is that near the interface the caustic should not be expected to be duplicated precisely by the computational results.

CRACK APPROACH TO THE INTERFACE

In our attempts to generate a crack at the interface after manufacture of the bi-material specimens it was observed that it is not an easy or automatic matter to make a crack propagate to the interface. In fact, considerable manipulation appeared to be required; this statement seems to hold at least under the present constraints on equipment which does not allow us to control the direction of crack propagation through a biaxial loading scheme. Instead, it was necessary to force the crack to propagate by hand manipulation to approach the interface. An example of typical crack paths is shown in Figure 12 where the direction of crack propagation is indicated by the arrows. The magnification of the photographs is about 30x so that the distance of closest approach of the crack to the interface is determined as 0.25 mm. With a future study which draws on construction of new equipment that allows control of biaxial straining it will be possible to examine the strict path dependence of cracks in the vicinity of an interface.

Eventually we found it possible to force the crack through (manual) manipulation to the interface. This procedure did not allow any evaluation of a criterion for the conditions of growth towards and into the interface. However, it allowed us to make specimens for studying crack growth along the interface. These investigations are delineated in the following section on crack propagation along the interface.

Figure 12. Crack path avoiding interface.

CRACK PROPAGATION ALONG THE INTERFACE

Having learned to force a crack to the interface, specimens were strained in the manner illustrated for the testing of the 65/35 formulation described in the section entitled "Design of the Interface Fracture Specimen", (see also Figures 6 and 7).

Usually a part of that crack propagation process involved the formation of a branch away from the interface. In this context we refer to a branch as the crack after it propagates away from the interface. The reason as to why such branching occurred is not very well established, though we have a tentative explanation: we have noted in the section entitled "Crack Propagation in Monolithic Viscoelastic Solids", that the interface, though cast into the specimens in a "virgin state", may not represent the same molecular constitution across the interface as one would expect in a material all cast from one monolithic piece. Expressed in another manner, it would seem reasonable that the interface strength in the bimaterial solid is less than the intrinsic strength of either of the two solids.

In order to interprete the consequence of this lower interface strength on the crack propagation behavior we need to recall that the growth of a crack at an interface is given, for plane stress, by the relation for the stress intensity factor K and crack speed $\dot{c}$

$$\frac{1}{2}\left\{D_1(\frac{\alpha}{\dot{c}}) + D_2(\frac{\alpha}{\dot{c}})\right\}K^2 = \Gamma \; ; \qquad \alpha \doteq 3\,\frac{\pi K^2}{8\sigma_0^2} \tag{2}$$

D_1 and D_2 are the creep compliances of the two adhering solids and Γ is the interface strength betrween the two solids and α a length parameter on the order of a small cohesive zone size and given by the equation shown in which σ_0 is the maximum stress carried across the interface. The corresponding relation for crack growth in either one of the two solids would be

$$D_1(\frac{\alpha}{\dot{c}})K^2 = \Gamma_1 \; ; \qquad \alpha \doteq 3\,\frac{\pi K^2}{8\sigma_{01}} \tag{3}$$

and

$$D_2(\frac{\alpha}{\dot{c}})K^2 = \Gamma_2 \; ; \qquad \alpha \doteq 3\,\frac{\pi K^2}{8\sigma_{02}} \tag{4}$$

for materials 1 and 2, respectively, where Γ_1 and Γ_2 are the appropriate intrinsic fracture energies and σ_{01} or σ_{02} the maximal stress for the two materials. Detailed examination of these equations shows that if all three fracture energies were the same then the crack propagation speeds for the interface separation should fall between those of the two solids at the same strain level: in the present test configuration this means that the stress intensity factor K would be the same, provided the material has been stressed long enough to allow achievement of the long term or rubbery modulus.

It is appropriate at this time to remind ourselves that the tests were conducted under the assumption that the mechanical properties of the Solithane compositions as well as the crack propagation behavior of one of these (namely 50/50) was known from previous studies. This assumption was made because to completely re-characterize these materials would have been more time consuming

than was deemed necessary at the outset of the program. At the same time it must be recognized that the supplier of these materials may have changed the chemistry of the material components without making that fact known. Thus some of the fracture behavior may have changed since the earlier studies devoted to the fracture of these model materials.

In fact, it should be noted that the long term relaxation modulus of the compositions of Solithane 113 was found to be independent of the composition; specifically the rubbery modulus for the 50/50 composition was the same as that for the 35/65 one (cf. Figure 3). This observation would lead one to believe that the intrinsic fracture energy might also be the same for these two materials. This conjecture was, however, not confirmed in the crack propagation studies. That fact follows from a comparison of the crack propagation data illustrated for the 50/50 composition in Figure 2 and in Figure 6 for the 35/65 composition: The asymptotic strain values for slow to zero crack propagation are not the same for these two compositions. In this statement we are aware that the data for the 35/65 composition is not fully present, although all the indications from the data -tempered by our understanding and experience in this field- indicate that the asymptotic strain for the 35/65 composition is higher than that for the 50/50 composition, almost by a factor of two.

In Figure 13 are reproduced the crack propagation data for the two monolithic compositions as well as the data for crack propagation along the interface. Note that the data are all related to 23°C, the temperature adjustment for the 35/65 composition being made on an extrapolated basis of the data in Figure 5*. One observes first that according to equation (2) the dependence of the interfacial crack propagation speed should have the same or similar characteristics as that for the individual materials considered by themselves. The basic reason for this expected similarity is that the crack propagation speed is determined by the creep compliance of the material, which is similar for the two materials used in this study (compare the relaxation moduli for the two materials in Figure 3). While the data for interfacial crack propagation does not cover as complete a strain or velocity range, it is clear that the slope of the data on the plot is indicative of this similarity.

We note further that the interfacial crack propagation data indicates faster growth rates than for either material alone. There are two possible reasons for this, though one is judged to be a more compelling one: We start with the less likely explanation. In this regard we first note that in Figure 3 the long time or rubbery modulus of the two Solithane compositions used here are the same. Therefore, if the strain has been applied to the specimen made of both of these materials we would expect that after sufficient time both halves of the materials in the two-material interfacial specimen have relaxed to the same modulus and thus renders a stress field at the crack tip that is the same as that when either materials are considered separately. It was in fact true that the interface fracture data represented in Figure 13 was derived from measurements that occurred over such long times that this relaxation should have occurred. If that condition was not satisfied to the degree we believe to be true, then the stress intensity factor -proportional to the strain- would have been actually higher than indicated; the result of this possibility is that the interfacial crack propagation speed should have been plotted at an effectively higher strain level than shown. Such should be the case, however, only if the rubbery modulus of the 35/65 composition is higher than shown in Figure 3. In other words, such should be the case only, if the supplier of the raw materials (Thiokol) has changed their chemical composition.

The other reason, and the more rational one under the present circumstances, is that the intrinsic strength of the interface is lower than that of the materials by themselves. That this is a logical explanation is, in retrospect, reasonable and consistent with microscope observations on interfacially failed specimens. Recall that the specimens were manufactured in a process in which one of the components

* The data in Figure 5 was time-temperature shifted and the shift factor extrapolated to 23°C.

was cast and partially cured before the other component was added. In this partial cure process the surface of the first component retained few chemically active sites so that fewer bonds across the interface were generated than when a single is material cast in monolithic form. This situation is, in fact, very similar to the manufacturing process of solid propellant rocket motors in which one material is deposited on another after the first one has been placed.

The optical appearance of the interface supports this view. The composite micrograph in Figure 14, of interest also in connection with the later discussion of branching away from the interface, represents the fractured interface across the whole specimen thickness. It shows two types of surfaces: one a specular or smooth surface section with intersecting lines like waves, and another section which exhibits finely textured lines running the length of the interface. These latter lines are the result of machine marks from the mold against which the silicone dam was made, and against which dam the Solithane was then cast. The fact that the cast surface is so well preserved indicates that the interface conditions did not produce a dense chemical connection across it. In all situtations where separation occurred along the interface this type of appearance was observed.

We may estimate the degree of bond reduction from the available data. Let us follow the lead of equation (2) which indicates that the time or velocity scale is governed by the creep compliance while the stress intensity scale (or strain scale in this case) is governed by the value of the intrinsic interfacial fracture energy. Thus a multiplicative factor on the stress intensity or the strain shifts the strain-velocity curve along the vertical logarithmic axis. That shift has been indicated in Figure 13 in terms of the data points identified by "VS" (vertically shifted). We note that in this way the interfacial fracture data can be brought into agreement with the monolithic fracture data if one allows that the interfacial strength is only about one half of the intrinsic average strength of the two materials. This value of the interfacial fracture strength is quite reasonable and indicates that, unless special chemical processing conditions are followed, it is not likely that the intrinsic material strength is achieved in a bonding process.

The ideal way in which the interfacial fracture energy is determined is to conduct the crack propagation experiments at elevated temperatures for long periods of time. Under these conditions the time dependent material behavior (viscoelasticity) is virtually inoperative and (near) elastic conditions prevail so that the fracture energy can be determined without interference or uncertainty due to the viscoelastic response in the materials. These measurements could not be made in the available time frame because the temperature equipment was not functioning properly.

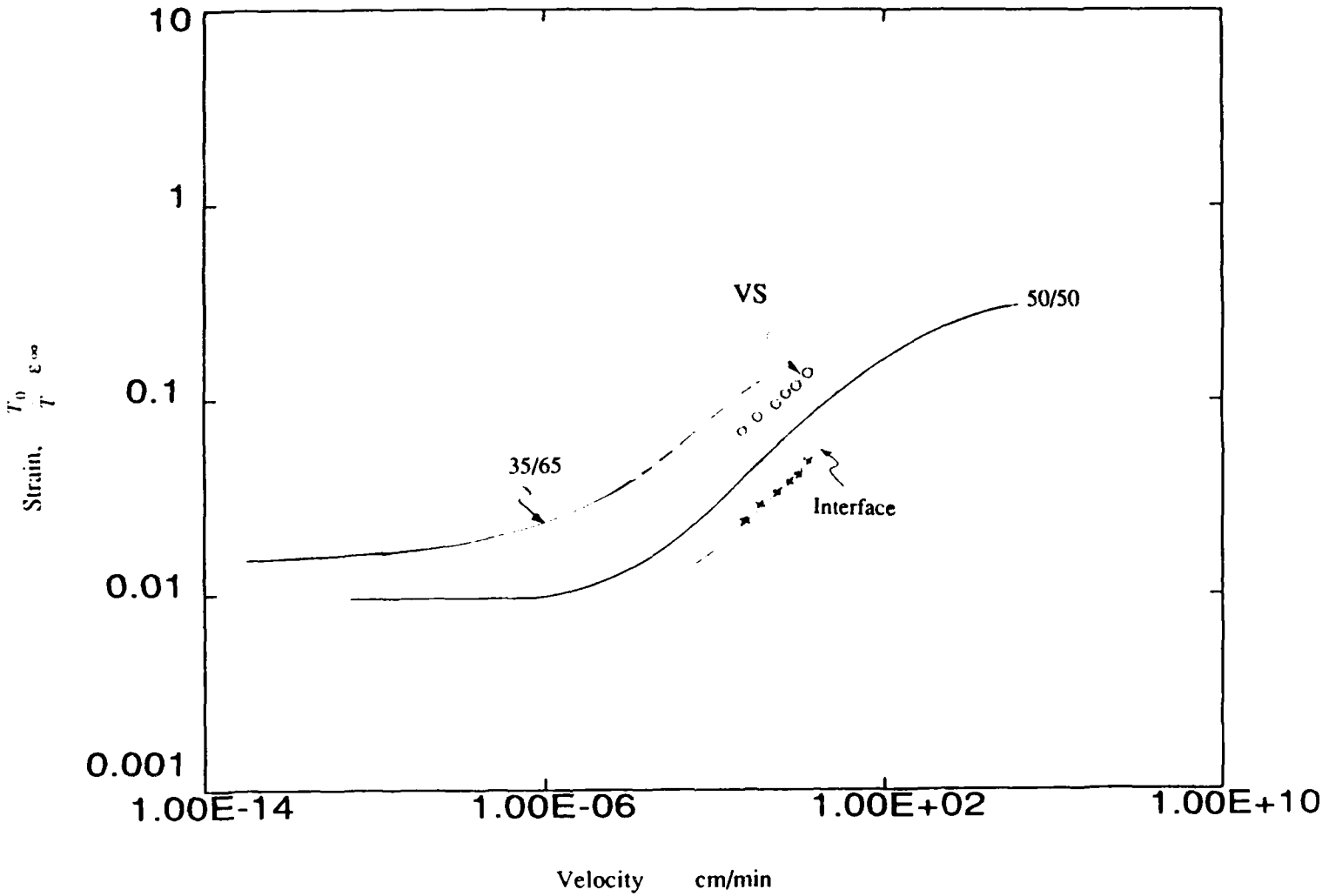
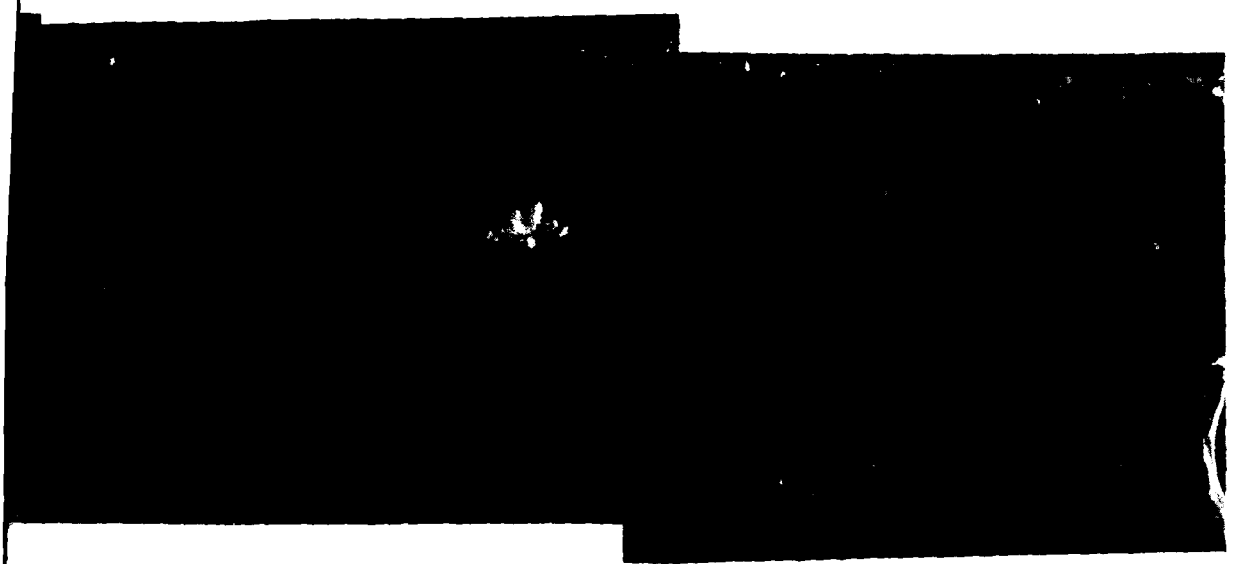


Figure 13. Comparison of crack growth along interface with that in either adherends (23°C).

Figure 14. Micrograph (approximately 30x) of interface separation and near-interface fracture in 50/50 composition.

[redacted]



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CRACK BRANCHING AWAY FROM THE INTERFACE

There are several issues connected with the topic of crack branching away from the interface, namely the condition that determines whether a crack will branch away, the angle at which the branched crack will propagate from the interface and the time required for the branching to be complete. We have, at this time no definitive information as to the condition(s) that result in branching, (criterion for branching). We have made observations and measurements regarding the branch angle as well as some regarding what governs the time that is required for the process to be completed.

We refer again to Figure 14 and specifically to the portion that identifies the direction of crack propagation by the arrow shown. Near that arrow an irregular surface feature indicates the onset of branching which occurred always into the 50/50 composition which is the "softer" of the two formulations. It appears that for as yet unknown reasons a strong bond between the two solids had been formed locally which allowed tearing into the softer of the two formulations. This type of appearance was quite uniform whenever branching occurred.

The fact that the crack propagated into the softer of the two materials could be determined readily through high magnification optical microscopy: Under high magnification the depth of field is quite shallow; in order to focus on portions of the surface having different elevation it is necessary to refocus the microscope and in this process it is possible to determine whether one or the other surface is located higher or lower with respect to other.

In the case of Figure 14 the crack did not branch away from the interface with an angled crack path. The reason for this is apparently that the "strong spot" extended only over a portion of the surface. Once propagation into the softer material had started then the adjacent weak interface determined that the crack path in the softer material stay close to the interface. Apparently, branching occurs successfully only if such a strong spot extends almost or all the way across the interface.

It is noteworthy to also point out in this context that disturbances at or near the interface such as bubbles did not apparently play a role in the branching process, even if these disturbances were, what one might call, major. For example, we show in Figure 15 the passage of the crack along a portion of the interface where it encountered several bubbles that were attached to the lateral surface of the sheet specimen in the interface region, although there is some indication that just prior to the successful branch there was a branch attempt at one of the small bubbles that is located on the near-viewing side of the sheet. However, several large bubbles, -dimly visible along the prebranch section of the interface and out of focus on the "other" side of the specimen- did not appear to influence the growth. Another example of this situation is rendered in Figure 16. Figure 17 shows an example of branching akin to that in Figure 14 where the branch starts apparently on a portion of the interface (in the first and second of the photos of that figure) and then becomes successful. Figure 18 shows a branch that -within the limited number of tests that lead to branching- is typical of a branch appearance: the branch tip is slightly irregular and not smooth so that the side view of Figure 14 is suggested.

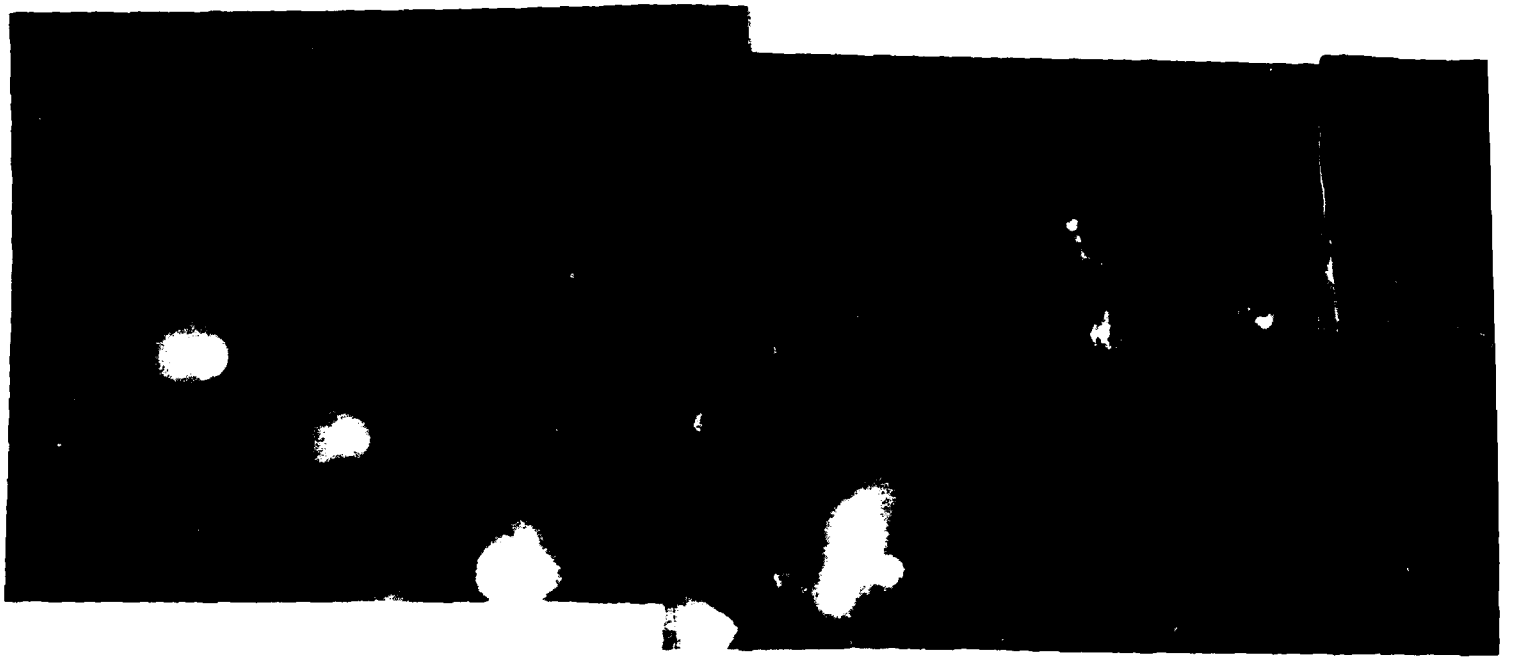


Figure 15. Interface-near crack path showing that bubble distances in stress field did not influence crack path.

[redacted]

[redacted]

Figure 16. Material irregularities (bubbles) do not effect branching (in a primary way).

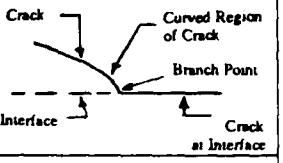
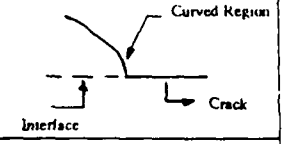
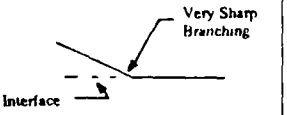
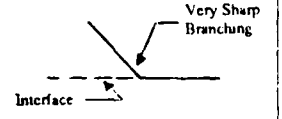
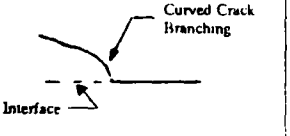
Figure 17. Example of branching.

Figure 18. Example of successful branching process.

Branch Angles:

The angles of branching were measured as well as possible with the irregular appearing surfaces. When branching occurred at the strain levels used in these experiments, the branch angle was relatively constant and on the order of 6 degrees. The accompanying Table 1. gives particulars of the angle; there seems to be no particular correlation between the angle and the strain level, at least not at the strains enountered here.

TABLE 1. - BRANCH ANGLE DATA

Specimen No.	Strain History	Branch Angle	Comment
2	5%	6°	
5	5%	8° tangent to initial crack direction 6° slope at a later line	
6	6%	5°	
9	4% 2%: crack branches when strain reduced to 2%	32°	
3	5%	12°	
11	3%	about 4°	test terminated before appreciable crack propagation beyond the branching point

An interesting phenomenon, but not well established because of the limited number of runs in this regard, has to do with the effect of load history on the branch angle. On one specimen the strain was reduced from 4% to 2% during crack propagation in order to obtain additional crack speed data at a lower strain level, (lower stress intensity). It so happened that in connection with the change in strain there was observed branching, and that branching occurred with a large angle. There appears to be no particular reason why branching should have occurred at that "moment", except that the strongly viscoelastic behavior of the 35/65 composition would certainly introduce a pronounced change in the crack tip stress field which could account for the marked deviation from the otherwise observed behavior.

Incubation Time for Branching:

Branching is apparently a phenomenon that requires a certain time to be accomplished. This observation is reasonable in light of the apparently irregular surface geometry associated with the branching process, namely the fact that branching does not occur along a neatly defined line across the specimen thickness. In passing from an interface crack to the fully developed and branched crack established at some distance away from the interface requires time. This time is taken in forming a new, more or less straight crack front in the off-side material, after the crack has propagated along a similarly straight front along the interface. Figure 19 shows a record of crack tip position as a function of time. We note that at first the crack propagates with a relatively high velocity of 0.206 along the interface and then, as it encounters the disturbed geometry of the branch process, slows considerably to about 0.028 cm/min. or, on the average 0.035 cm/min., before propagating at a larger and steady velocity of 0.05 cm/min. This phenomenon is observed consistently with the branching process and is understandable on primarily the basis of the irregularity of the topology of the initial branch geometry. It has not been possible, however, on the basis of these limited number of tests to establish a quantitative assessment of the branch incubation time with the applied strain or with the branch geometry. material of a given thickness. Holes are placed into the platens for filling the mold and for allowing escape of air during molding. Actually, the mold is filled by sucking the Solithane into the mold cavity through the application of a vacuum.

In order to produce bi-material sheets it was first tried to fill the mold partially (half way) with the mold faces aligned vertically; that portion of Solithane was then cured lightly so that mixing with a second charge of different composition could not take place. It developed that a meniscus formed in the process of the first half-casting; this meniscus was was too pronounced to yield an even approximately flat interface as viewed across the thickness coordinate of the sheet.

Specimens with this meniscus interface were useful only in studies in which the crack approached the interface at some angle.

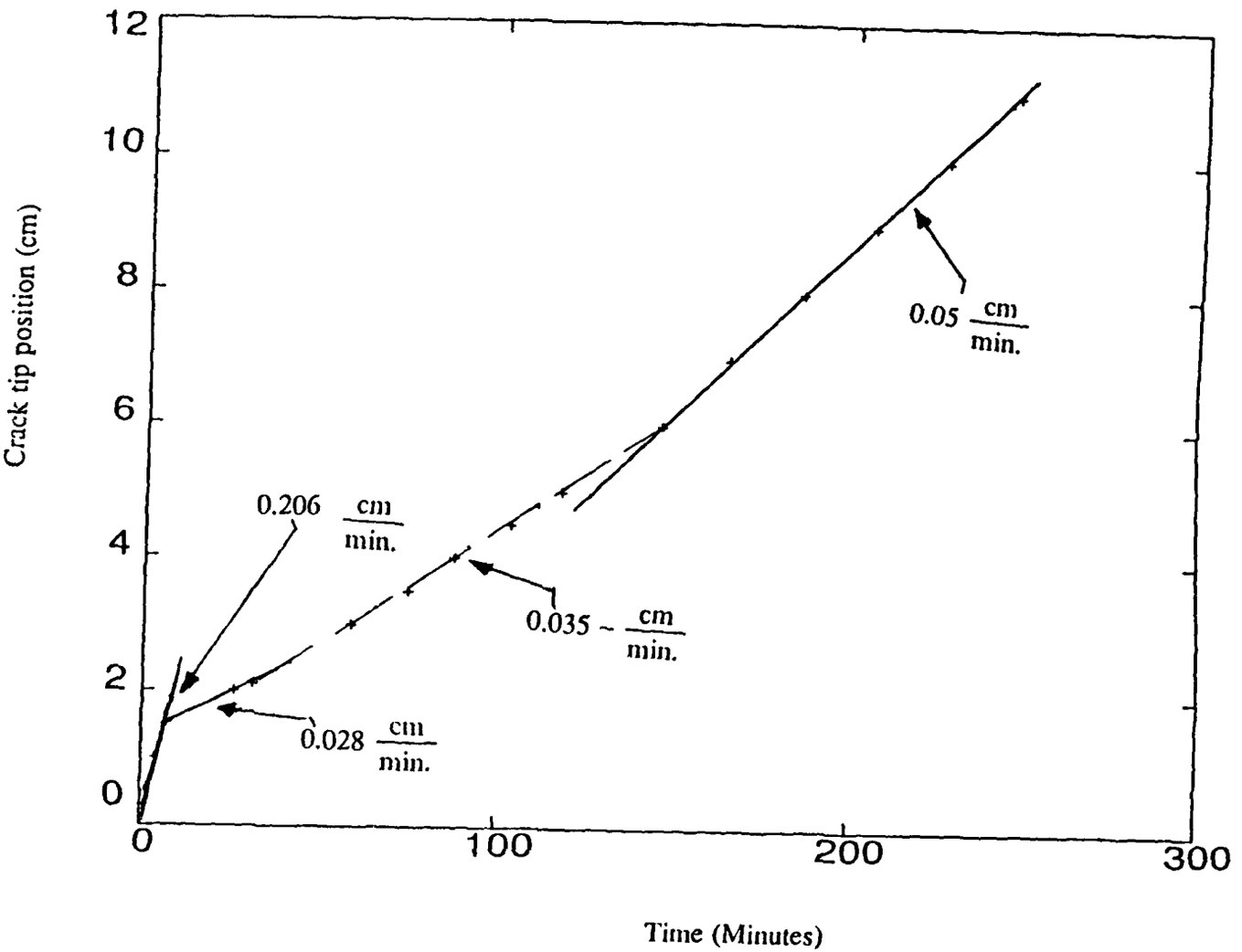


Figure 19. Crack-tip position vs time in a branching process.

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2. Knauss, W.G., "Fracture Mechanics and the Time Dependent Strength of Adhesive Joints," GALCIT SM Report 70-6, 1970. *J. Comp. Materials*, Vol. 5, p. 176, 1971.

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